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ORIGINAL ARTICLE

Effects of land-use change on chemical composition of soil organic matter in tropical lowland Bolivia

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Abstract

Land-use change affects not only the amount of soil organic matter (SOM) but also its composition. We performed cross-polarization magic angle spinning (CPMAS) ^{13}C and ^{15}N nuclear magnetic resonance (NMR) spectroscopy to investigate the chemical composition of bulk SOM in topsoils (0–15 cm) under different land use, namely native forest (NF), 27-year cropland with wheat/soybean rotation (CL) and 27-year rangelands with guineagrass (RG) and with bahiagrass (RB), in south-east Bolivia. The findings of this study showed only a subtle alteration of composition of bulk SOM despite the large changes in carbon (C) content. Nevertheless, NF and RB showed a slightly lower abundance of aromatic C but a higher proportion of alkyl C compared to CL and RG where the loss of organic matter was substantial. This suggests that relatively stable components dominated by aromatic structures had relatively enriched during SOM decomposition under agricultural practices. A slight disparity of SOM composition observed between RG and RB (less O-alkyl C but more aromatic C in RG than RB) suggests that grass species influenced SOM quality even under the same land use, namely the rangeland. On the other hand, organic N composition was less affected by land use or management practice than C forms.

Introduction

It is known that there is a dynamic balance between organic input and output in tropical forest ecosystems (Greenland *et al.* 1992). However, this balance is very fragile and susceptible to changes in land-use and/or management practice (Lal and Kang 1982; Tiessen *et al.* 1994). The clearance of tree cover (i.e. deforestation) often leads to a rapid decline in soil organic matter (SOM) resulting in land degradation and enhanced carbon dioxide emission (Detwiler 1986). In general, the risk of SOM loss is higher in tropical zones than in temperate regions due to the elevated C turnover in the tropics (Jenkinson and Ayanaba 1977; Tiessen *et al.* 1994). There are numerous studies showing a rapid decline in organic C content in cultivated soils of the tropical regions (Detwiler 1986; Greenland *et al.* 1992; Guo and Gifford 2002). Despite that both quality and quantity of SOM have a major influence on SOM dynamics, much fewer studies were conducted on qualitative alterations of SOM imposed by land-use change and/or management practice (Theng *et al.* 1989; Greenland *et al.* 1992). Moreover, there is apparently less information available in tropical regions as compared to temperate areas, especially when advanced analytical tools are implemented, for example solid-state nuclear magnetic resonance (NMR) spectroscopy.

A typical case of soil degradation has been documented in Colonia Okinawa, south-east Bolivia (Abe *et al.* 2007) where the large-scale mechanized farming system and extensive livestock (mainly cattle) production has been developed without the use of fertilizers since Japanese immigrants started to settle in 1956 (Takata *et al.* 2003). In this region, Abe *et al.* (2007) reported a significant decline in C, N and P levels and Mehlich-3 extractable P, Ca and Mg in the soils after conversion of semideciduous forests to agricultural

lands. The authors also found close correlations between these components which strongly affected the SOM dynamics.

In the given study, we tried to evaluate the extent to which SOM composition of bulk soils was altered due to the land-use change. Therefore, we assessed organic forms of C and N in bulk SOM under different land use by solid-state ^{13}C and ^{15}N NMR spectroscopy.

Materials and methods

Study site and soil samples

The general description of the study site was previously documented by Abe *et al.* (2007). In summary, the study site (17°21′S, 62°47′W) is located on an alluvial plain in Colonia Okinawa-II, Santa Cruz, south-east Bolivia. The soils were classified as Fluventic Haplustepts (Takata *et al.* 2003). The area has a mean annual temperature of 23.8°C, mean annual precipitation of 1275 mm and mean annual evaporation of 1619 mm (Hitsuda *et al.* 1996). We examined three types of land uses: (i) primary forest (NF) which was often dominated by *Gallesia integrifolia*, *Anadenanthera macrocarpa*, *Astronium* cf. *fraxinifolium* and *Enterolobium contortisiliquum*; (ii) 27-year old cropland (CL) where wheat (*Triticum aestivum*) and/or soybean (*Glycine max*) have been cultivated twice a year; and (iii) 27-year old rangelands with a grazing rate of approximately 1.5 cattle ha^{-1}. The rangeland plot was further divided into two subplots according to grass species, namely guineagrass, *Panicum maximum* (RG) and bahiagrass, *Paspalum notatum* (RB). The land use selected in this study is prevalent in the region and the farming period of 27 years roughly corresponds to the average duration of land cultivation in the study area. The study site has received neither fertilizers nor crop residues since the forests were cleared in 1975. Ten soil samples were collected from the Ap horizon (0–15 cm) or equivalent layers at each plot or subplot as described by Abe *et al.* (2007). These samples were subjected to air-drying, gentle grinding and sieving (2-mm mesh). Visible plant fragments and charcoals were removed by forceps as much as possible. Afterwards, a subset of the samples were mixed to make a composite sample for each plot/subplot. The soil samples were free of carbonates according to the HCl test and, hence, soil C was considered to exist thoroughly in organic forms. Selected physicochemical properties of these samples are shown in Table 1. The analytical method of these properties was described by Abe *et al.* (2007) except for the total C, N and S concentrations which were determined in duplicate by dry combustion with an elemental analyzer (Vario EL or Vario MAX CNS; Elementar, Hanau, Germany). In this study, we evaluated the loss of SOM under different land use by calculating the soil organic C saturation deficit according to Van Noordwijk *et al.* (1998). Assuming the soil C content under natural forest (NF) as representative of the initial C contents at the study site, soil organic C deficits were in the order of –58%, –41% and –10% in CL, RG and RB, respectively (Table 1). To the same extent, the total N content of the soils changed by –59%, –41% and –14% in the order of CL, RG and RB, respectively. These declines in C and N were well associated with phyto-available (Mehlich-3 extractable) nutrients such as Ca, Mg, P and Zn (Abe *et al.* 2007).

Table 1 Selected physicochemical properties of topsoil (0–15 cm) samples under different land use at the study site

		Plot			
Property	Unit	NF	CL	RG	RB
Total C†	g kg^{-1}	24.1	10.2	14.1	21.8
Total N†	g kg^{-1}	2.90	1.20	1.70	2.50
Total P‡	g kg^{-1}	0.45	0.33	0.34	0.41
Total S†	g kg^{-1}	0.50	0.30	0.30	0.40
Mehl. P§	mg kg^{-1}	86.0	18.0	26.8	37.2
Mehl. Ca§	cmolc kg^{-1}	2.91	1.27	2.01	2.16
Mehl. Mg§	cmolc kg^{-1}	1.23	0.58	0.87	0.98
Mehl. K§	cmolc kg^{-1}	0.50	0.90	0.56	1.04
Mehl. Mn§	g kg^{-1}	1.06	0.82	1.15	1.38
Mehl. Fe§	mg kg^{-1}	160.9	166.1	201.3	243.7
Mehl. Cu§	mg kg^{-1}	1.9	1.5	2.3	2.5
Mehl. Zn§	mg kg^{-1}	6.0	2.8	3.4	5.3

†Dry combustion method (Vario MAX CNS); ‡Wet digestion method; §Mehlich-3 extraction method (Mehlich 1984); NF, native forest; CL, 27-year cropland with wheat/soybean rotation; RG, 27-year rangelands with guineagrass; RB, bahiagrass.

Hydrofluoric acid treatment prior to NMR analysis

Subsamples of the composites were ball-milled and treated with 10% (v/v) hydrofluoric acid (HF) as described by Spielvogel *et al.* (2008). The HF treatment improves NMR spectra by increasing signal to noise ratio due to the enrichment of C and N and removal of paramagnetic minerals in the soils, without major alteration of composition of organic C and N detectable by NMR analysis (Schmidt *et al.* 1997; Gonçalves *et al.* 2003).

Solid-state ^{13}C and ^{15}N NMR analysis

Solid-state NMR measurement was performed by employing the cross-polarization magic angle spinning (CPMAS) technique. CPMAS ^{13}C NMR spectra were recorded at a resonance frequency of 50.3 MHz, rotation frequency of 6.8 kHz, contact time of 1 msec and a delay time of 200 msec (DSX 200; Bruker, Rheinstetten, Germany). The chemical shift of ^{13}C NMR spectroscopy was given relative to

Table 2 Recovery and enrichment of C and N in topsoil (0–15 cm) samples after the treatment with 10% hydrofluoric acid (HF)

Entry	Unit	Plot			
		NF	CL	RG	RB
Total C†	%	2.4	1.0	1.4	2.2
Total N†	%	0.29	0.12	0.17	0.25
C/N ratio	–	8.3	8.5	8.3	8.7
Weight loss	%	87.5	91.6	89.3	87.3
Total C after HF‡	%	17.2	10.3	10.8	14.3
C Recovery	%	87.8	84.8	81.6	83.3
C Enrichment	%	702	1007	765	656
Total N after HF‡	%	1.77	0.96	1.04	1.38
N Recovery	%	76.8	65.5	65.7	71.5
N Enrichment	%	614	777	616	563
C/N ratio after HF	–	9.7	10.7	10.4	10.4

†Dry combustion method (Vario MAX CNS); ‡Dry combustion method (Vario EL); NF, native forest; CL, 27-year cropland with wheat/soybean rotation; RG, 27-year rangelands with guineagrass; RB, bahiagrass.

tetramethylsilane (0 p.p.m.) and was adjusted with glycine (176.04 p.p.m.). CPMAS ^{15}N NMR spectra were acquired at a frequency of 40.5 MHz, rotation frequency of 5.7 kHz, contact time of 0.7 msec, and delay time of 200 msec (DMX 400; Bruker). As reference for the chemical shift, nitromethane was taken (0 p.p.m.) for ^{15}N NMR spectroscopy. Possible assignments of resonances in ^{13}C and ^{15}N NMR analysis were referred to Kögel-Knabner (1997). The relative abundance of organic C was calculated based on the integrals of alkyl C (–10–45 p.p.m.), O-alkyl C (45–110 p.p.m.), aromatic C (110–160 p.p.m.) and carbonyl C (160–220 p.p.m.). We did not account for spinning side bands. In addition, the alkyl C/O-alkyl C was calculated as potential indicators for the degree of decomposition (Baldock *et al*. 1997). It is believed that the decomposition index increases during the decomposition of natural organic materials (Baldock *et al*. 1997).

Results

Recovery and enrichment of C and N after HF treatment

Table 2 shows changes in total contents of C and N after HF treatment. The recovery of C (81.6–87.8%) was higher than that of N (65.5–76.8%), which resulted in higher values of C/N ratio (9.7–10.7) after HF treatment than before the treatment. Both rates of recovery for C and N were somewhat higher in NF than CL, RG and RB. Total contents of C and N in the samples were considerably enriched by 656–1007% and 563–777%, respectively.

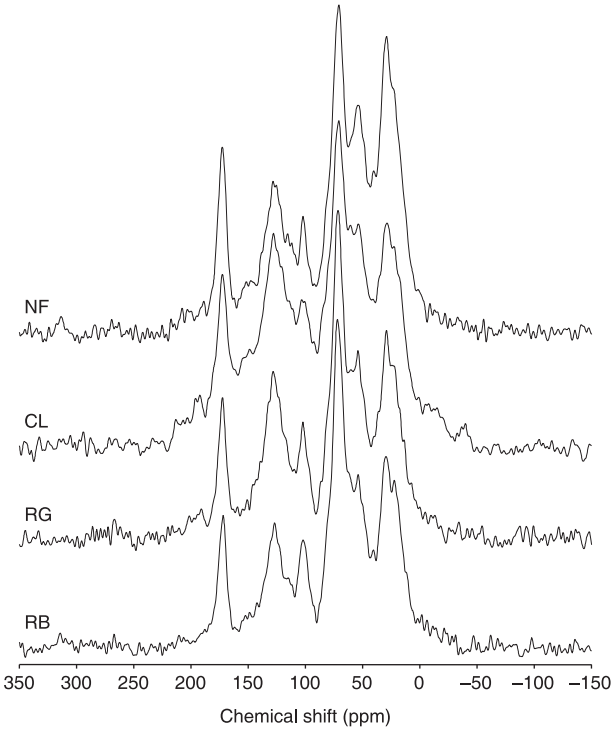

Figure 1 Cross-polarization magic angle spinning (CPMAS) ^{13}C and ^{15}N nuclear magnetic resonance (NMR) spectra of hydrofluoric acid (HF)-treated topsoil (0–15 cm) samples under different land use at the study site. NF, native forest; CL, 27-year cropland with wheat/soybean rotation; RG, 27-year rangelands with guineagrass; RB, bahiagrass.

CPMAS ^{13}C NMR analysis

Cross-polarization magic angle spinning ^{13}C NMR spectra of the HF-treated samples are given in Figure 1. Methylene structures dominated alkyl C as indicated by the peak at 29 p.p.m. A major portion of O-alkyl C is assignable to polysaccharides indicated by the distinct signal centered at 72 p.p.m. in combination with the resonance at 105 p.p.m. Methoxyl structures (resonance at 55 p.p.m.), which may be derived from lignin as well as N-alkyl from amino acids, are also contributing to the O-alkyl C region between 45 and 60 p.p.m. Phenolic C and C-substituted aromatic C are contributing to the chemical shift region between 140 and 160 p.p.m. Carbonyl C (resonance at 175 p.p.m.) includes carboxylic C, carbonylic C and/or amide-C in various compounds.

Table 3 shows the relative contribution of organic C as revealed from NMR spectra. Organic C at the study site roughly consisted of 40% of O-alkyl C, 30% of alkyl C, 20% of aromatic C and 10% of carbonyl C. Nevertheless, the CL soil apparently contained less alkyl C (25.9%) but more aromatic C as compared to the NF soil. The RG soil showed

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Table 3 Integrals of the ^{13}C nuclear magnetic resonance (NMR) chemical shift regions from topsoil (0–15 cm) samples after the treatment with 10% hydrofluoric acid (HF)

Plot	Alkyl C (–10/45 p.p.m.)	O-alkyl C (45/110 p.p.m.)	Aromatic C (110/160 p.p.m.)	Carbonyl C (160/220 p.p.m.)	Decomposition index†
NF	31.1	40.8	17.1	10.9	0.76
CL	25.9	41.4	21.8	10.8	0.63
RG	25.9	43.6	20.5	9.9	0.59
RB	26.9	47.6	16.5	9.1	0.57

†Decomposition index = alkyl C/O-alkyl C. NF, native forest; CL, 27-year cropland with wheat/soybean rotation; RG, 27-year rangelands with guineagrass; RB, bahiagrass.

a very similar organic C composition to the CL soil, whereas the RB soil had less alkyl C but similar aromatic C as compared to the NF soil. In the soils of RG and RB, relatively more O-alkyl C was observed than under forest and cropland (NF, CL). No distinctive differences were detected for carbonyl C between the sites. A decrease of the decomposition index was found in the order of NF >> CL > RG ≈ RB.

CPMAS ^{15}N NMR analysis

Cross-polarization magic angle spinning ^{15}N NMR spectra of HF-treated bulk soil samples are shown in Figure 2. The soil material from CL was excluded from ^{15}N NMR analysis due to the very low N content (below 1%). For ^{15}N-NMR N contents larger than 1% are crucial to obtain acceptable signal to noise ratios within an acceptable number of scans (Knicker *et al.* 1993; Knicker and Lüdemann 1995). Even for the samples with N contents more than 1% after HF pretreatment, the quantification of N forms was not possible due to the low signal to noise ratio of the spectra. All CPMAS ^{15}N NMR spectra were predominantly characterized by a signal at –259 p.p.m. (Figure 2), which indicates amide-N of peptide structures as the major organic N form. The shoulder of the signal at –259 p.p.m. between –185 and –245 p.p.m. (pyrrole/nitrile-type N) in RG suggests the presence of heterocyclic aromatic N. In addition, there was also a significant but small signal at –347 p.p.m. in RG corresponding to free amino groups attached to amino acids and amino sugars.

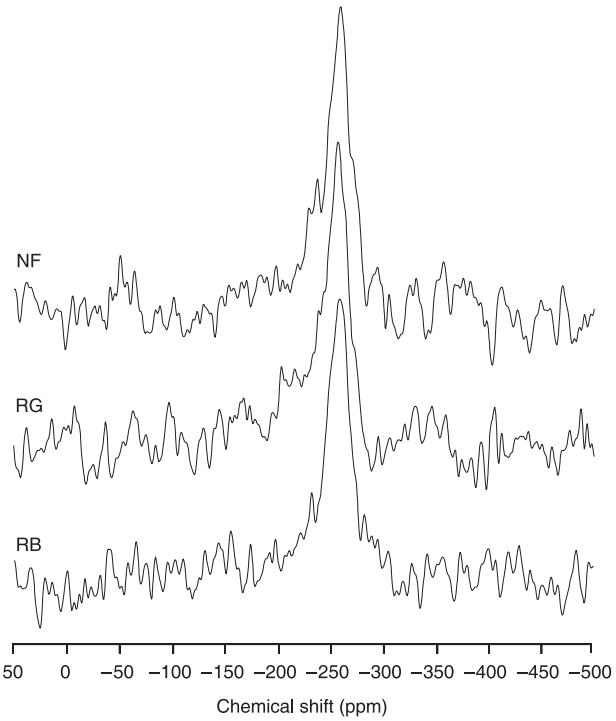

Figure 2 Cross-polarization magic angle spinning (CPMAS) ^{15}N NMR nuclear magnetic resonance (NMR) of hydrofluoric acid (HF)-treated topsoil (0–15 cm) samples under different land use at the study site. NF, native forest; RG, 27-year rangelands with guineagrass; RB, bahiagrass.

Discussion

Preferential recovery of C to N after HF treatment (Table 2) is often found in soils from various regions (Schmidt *et al.* 1997; Gonçalves *et al.* 2003; Diekow *et al.* 2005a). This might be attributed to soil mineral properties (in particular, soil texture and mineralogy), organic matter characteristics and the degree of organo–mineral associations because C and N soluble in HF can be regarded as mineral-associated fraction (Spielvogel *et al.* 2008). Therefore, the lower recovery rates of C and N in CL, RG and RB than NF suggest that agricultural practices such as mechanical tillage have disrupted soil structure and aggregations (Six *et al.* 1999, 2000).

Solid-state ^{13}C NMR analysis revealed that land-use change imparts only subtle alteration of the chemical composition of SOM despite large changes in its amount (Tables 1, 3). This result is basically the same as the previous studies which not only originate from the temperate region (Skjemstad *et al.* 1986; Hopkins *et al.* 1993; Fründ *et al.* 1994; Preston *et al.* 1994; Kinchesh *et al.* 1995; Mueller and Koegel-Knabner 2009)

but also from the tropical areas (Diekow *et al.* 2005a,b). On the other hand, there was a measurable disparity in soil organic C forms under different land use and even under different grass species in the rangeland (RG and RB). This suggests that various agricultural implementations after the clearance of natural forests can lead to alteration of SOM quality. We found increased proportions of aromatic C in CL and RG, where SOM depletion was substantial (Table 1). In contrast, aromatic C in RB, where soil organic C saturation deficit was little, was almost the same as NF. These results imply that aromatic C proportions increased with increasing decomposition stages of C. Stable compounds having aromatic structures can be enriched while other compartments are mineralized more rapidly due to the soil disruption by agricultural practices. These findings are comparable to a wide range of different soils (Mahieu *et al.* 1999; Diekow *et al.* 2005a, 2005b). Contrary to our expectation, the decomposition index was not relevant to soil C loss, namely organic matter decomposition, in this study. The highest decomposition index value regardless of its highest C content found in NF suggests a fast and vital cycle of organic matter dynamics in this tropical forest ecosystem. The lower values of decomposition index in RG and RB than NF, however, were responsible for the relatively higher values of O-alkyl C. This high contribution of O-alkyl C in RG and RB would be attributed to higher biomass production and cattle manure deposit. Fresh plant litter and cattle manure generally have high proportions of O-alkyl C (Chen *et al.* 1989; Diekow *et al.* 2005a). This clearly indicated that the decomposition index is not suitable for the assessment of land-use effects on SOM quality as different land use leads to different sources of organic matter with different chemical compositions. The use of the ratio between alkyl C and O-alkyl C can be restricted to soils with similar OM input and comparable decomposition kinetics (Baldock *et al.* 1997). We also detected a measurable difference in soil C forms between RG and RB, mostly expressed in the amount of aromatic C and O-alkyl C. The former can be attributed to enrichment of relatively recalcitrant compounds such as charcoal during soil C depletion, while the latter can be explained by differences in biomass production and cattle manure deposit depending on guineagrass and bahiagrass. This suggests that grass species affect both quantity and quality of SOM in the soil.

The solid-state ^{15}N NMR spectroscopic investigation revealed that the major part of the organic nitrogen is bound in amide-N functional groups, most probably as part of proteinaceous materials (Knicker *et al.* 1993). The predominance of amide-N is found to be a ubiquitous characteristic of organic N in many soils (Knicker *et al.* 1993; Knicker and Lüdemann 1995; Diekow *et al.* 2005b). The effect of land-use change on soil organic N was not conspicuous in the present study. This result is comparable to Dieckow *et al.* (2005b). Amide N derived from peptides, the major N form in both plants and microorganisms and their residues, can accumulate during humification. The reason why the land-use change induces no clear differences in soil organic N composition would be that the organic N form as amide and peptide N can still be introduced in spite of different land use.

Our findings showed that land-use change affects organic C forms but not organic N composition in the soils of the study region. Major trends of qualitative alteration of organic C observed in this study are: (i) increased proportion of aromatic C in CL and RG; and (ii) of O-alkyl C in RG and RB. The former can respond to preferential reaming of compounds having recalcitrant structure against microbial decomposition (e.g. charcoal), while the latter would be due to litter quality and cattle manure deposits. In terms of grassland management, grass species can influence not only organic C amount but also organic C forms in soils. These findings suggest that combined studies of SOM quality and quantity are needed to predict C cycling and to explore optimal C management in soils of diverse agroecosystems in the face of food insecurity and global warming.

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